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Vanadyl(acetylacetonate)₂ mediated hydrolytic splitting of 1,3,5-triazine in a solution of toluene at 130 °C: The crystal structure of its axial formamide adduct

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RESEARCH ARTICLE

ABSTRACT



A toluene reaction of vanadyl bis(acetylacetonate) with 1,3,5-triazine produces a symmetrical three-fold hydrolytic cleavage of the triazine, and these three formamide moieties are found in the crystal structure. One of the resulting formamides attaches itself to the sixth (axial) position of the vanadyl complex, producing materials in which the remaining two formamides are trapped in the resulting lattice. Those crystals belong in space group *Pca2₁*, *Z* = 4 and the final *R*-factor = 0.030 for 3213 data collected at 100 K.

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KEYWORDS

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 Vanadyl acetylacetonates
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SUPPLEMENTARY MATERIAL

PART I

There are some particularly attractive examples of formamides acting as ligands to various metals which we want to share with readers.

HUXPEY: Moon, D.; Choi, J.-H. Crystal structure of trans-di-chlorido-(1,4,8,11-tetra-aza-cyclo-tetra-decane-κ4 N)chromium(III) bis-(form-amide-κO)(1,4,8,11-tetra-aza-cyclo-tetra-decane-κ4N)chromium(III) bis-[tetra-chlorido-zincate(II)]. *Acta Crystallogr. E Crystallogr. Commun.* **2020**, *76*, 656–659.

<https://doi.org/10.1107/S2056989020004910>

C₁₂H₃₀CrN₆O₂³⁺, C₁₀H₂₄Cl₂CrN₄⁺, 2(Cl₄Zn²⁻) Sp. Gr. = *P2₁/n*, *Z* = 2.0, *Z'* = 0.5, *R* = 4.18, *T* = 220 K. Diffractometer, Av. Sig. = 0.001-0.006 Å. (**Figure S1**).

IBAYEQ: Palkina, K. K.; Orlova, V. T.; Smolentsev, A. Y.; Frolova, E. A. Coordination Compounds of Transition-Metal Nitrates with Formamide M^{II}I(NO~3)~2 · 2(HCONH~2) · 2H~2O (M^{II}I = Cu, Cd, Co, Mn). *Russ. J. Inorg. Chem.* **2004**, *49*, 157–161.

<https://www.tib.eu/de/suchen/id/BLSE:RN147815280/Coordination-Compounds-of-Transition-Metal-Nitrates?cHash=a5a6f7413765619ec97ce4219fbc5d8>

C₂H₁₀CuN₄O₁₀ Sp. Gr. = *P2₁2₁2₁*, *Z* = 4.0, *Z'* = 1.0, *R* = 3.90, *T* = 295 K, Diffractometer, Av. Sig. = 0.006-0.006 Å. (**Figure S2**)

IBAYIU: Palkina, K. K.; Orlova, V. T.; Smolentsev, A. Y.; Frolova, E. A. Coordination Compounds of Transition-Metal Nitrates with Formamide M^{II}I(NO~3)~2 · 2(HCONH~2) · 2H~2O (M^{II}I = Cu, Cd, Co, Mn). *Russ. J. Inorg. Chem.* **2004**, *49*, 157–161.

<https://www.tib.eu/de/suchen/id/BLSE:RN147815280/Coordination-Compounds-of-Transition-Metal-Nitrates?cHash=a5a6f7413765619ec97ce4219fbc5d8>

Diaqua-dinitrato-bis(formamide)-cadmium(II) Gr. = $P2_12_12_1$, Z = 4.0, Z' = 1.0, R = 3.90, T = 295 K, Diffractometer, Av. Sig. = 0.006-0.006 Å (Figure S3).

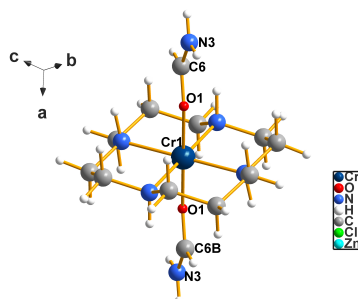


Figure S1. Bis(formamide)-(1,4,8,11-tetraazacyclotetradecane)-Cation.

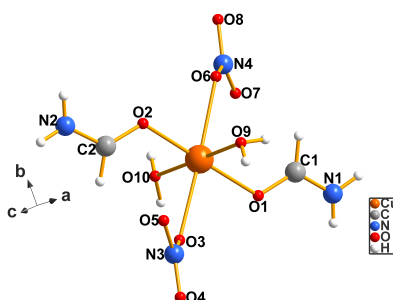


Figure S2. Diaqua-dinitrato-bis(formamide)-copper(II).

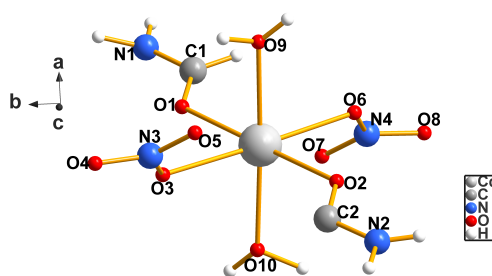


Figure S3. Diaqua-dinitrato-bis(formamide)-cadmium(II).

PART II

Historic records for triazine

1. Bis-triazinyls bipyridines (BTP's) have been proposed as useful in the reprocessing of nuclear wastes.

Reference: Hudson, M. J.; Drew, M. G. B.; Foreman, M. R. S.; Hill, C.; Huet, N.; Madic, C.; Youngs, T. G. A. The coordination chemistry of 1,2,4-triazinyl bipyridines with lanthanide(III) elements – implications for the partitioning of americium(III). *Dalton Trans.* **2003**, 1675–1685.

<https://doi.org/10.1039/B301178J>

The Abstract reads: “It has been established that 6-(5,6-dialkyl-1,2,4-triazin-3-yl)-2,2'-bipyridines (R,hemi-BTPs) have properties which are intermediate between those of the terpyridines and the bis(1,2,4-triazin-3-yl)pyridines (BTPs). However, they resemble the terpyridines much more closely than the BTPs. It has been shown that Et,hemi-BTP when dissolved in TPH—a dodecane-like solvent—is a selective reagent for the separation of americium(III) from europium(III). Solution NMR in acetonitrile largely confirmed the crystallographic results. There was no evidence for a 1 : 3 complex cation, or for significant differences between metal(III)–N distances for the pyridine and 1,2,4-triazine rings. Intramolecular hydrogen bonding plays a crucial role in the formation of metal coordination spheres, which explains the differences between the terpyridyl, R,hemi-BTPs and the BTPs. Protonation of the R,hemi-BTPs facilitates a conformational change which is necessary for complexation.”

2. Privileged s-Triazines: Structure and Pharmacological Applications

Reference: Shah, D. R.; Modh, R. P.; Chikhaliya, K. H. Privileged s-triazines: structure and pharmacological applications. *Future Med. Chem.* **2014**, 6, 463–477.

<https://doi.org/10.4155/fmc.13.212>

The Abstract reads: "This review summarizes recent reports on s-triazine and its respective analogs from the medicinal chemistry angle. Due to its high reactivity and binding characteristic towards various enzymes, s-triazine has attracted attention. This is combined with facile synthesis and interesting pharmacology. The triazine class demonstrates wide biological applications - including antimicrobial, antituberculosis, anticancer, antiviral and antimalarial. In this article the library of s-triazine-based molecular designs has been collated with respective bioactivity. Compounds are further compared with other heterocyclic/nontriazine moieties to correlate the efficiency of privileged s-triazine. We hope this article may assist chemists in their drug design and discovery efforts."



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